NOLTR 71-187

FABRICATION AND TESTING OF CARBON FIBER/PHENYLATED POLYQUINOXALINE RESIN COMPOSITES

By
Mary Lynda Santelli

18 NOVEMBER 1971

NOL

NAVAL ORDNANCE LABORATORY, WHITE OAK, SILVER SPRING, MARYLAND

NOLTR 71-187

FABRICATION AND TESTING OF CARBON FIBER/PHENYLATED POLYQUINOXALINE RESIN COMPOSITES

Prepared by:
Mary Lynda Santelli

ABSTRACT: Techniques were developed for fabricating carbon fiber composites using a Naval Ordnance Laboratory developed high temperature resin, phenylated polyquinoxaline (PPQ). Composites with interlaminar shear and flexural strengths about 80% of those for similar epoxy composites were obtained. Heat aging at 343°C (650°F), in air, revealed excellent initial strength retention at temperature but significant loss of strength after 168 hours.

APPROVED BY: F. Robert Barnet
Non-Metallic Materials Division
CHEMISTRY RESEARCH DEPARTMENT
NAVAL ORDNANCE LABORATORY
White Oak, Maryland

NOLTR 71-187 18 November 1971

Fabrication and Testing of Carbon Fiber/Phenylated Polyquinoxaline Resin Composites

Certain ordnance items require not only the stiffness, strength, and light weight offered by composite structural plastics, but also materials which can withstand high temperatures. A phenylated polyquinoxaline resin developed at the Naval Ordnance Laboratory (reference (14)) was reported to have thermal stability at high temperatures. This report covers work done under Independent Exploratory Development (IED) funding, from July 1969 to June 1971, to develop composite fabrication techniques using the phenylated polyquinoxaline resin and to test the resulting composites at high temperatures.

ROBERT WILLIAMSON II
Captain, USN
Commander

Albert Lightbody

ALBERT LIGHTBODY
By direction

CONTENTS

TABLES

ILLUSTRATIONS

INTRODUCTION

One of the many advantages of using carbon as a structural material is its stability at high temperatures (reference (1)). This property can only be utilized in carbon fiber-reinforced plastic composites to the extent to which the polymer matrix materials can withstand high temperatures. The upper short-term temperature limit for epoxy resins is about 177°C (350°F). One of the most stable commerically available resins, polyimide resin, has a maximum useful temperature in the 260 to 316°C (500 to 600°F) range depending on the strength retention and work life required (references (2)-(7)). Because of these limitations, work is constantly going on to develop laminating resins capable of withstanding even higher temperatures (references (8)-(13)).

A new series of high temperature resins called phenylated polyquinoxalines (PPQ) was developed at the Naval Ordnance Laboratory (NOL) in 1969. The chemical synthesis and evaluation of these resins is reported in reference (14). This report covers work done to develop suitable processing techniques for producing carbon fiber-PPQ composites and to evaluate their high temperature limitations. Simultaneously, similar work was done at the Whittaker Corporation, San Diego, California (reference (15)).

As a result of the first part of the work, successful fabrication techniques for flat laminates were developed. Carbon fiber/PPQ resin composites were fabricated with average room temperature shear and flexural strengths about 80% of those obtained with carbon/epoxy composites Void contents were in the 2 to 4% range. At high temperatures, the stability of the resin was found to depend greatly on its purity. For the purest resin, initial shear and flexural strengths of the composites at 343°C (650°F) were equivalent to those at room temperature. However, after 168 hours at 343°C in air and after only a 3% weight loss, the mechanical properties were found to have degraded significantly.

EXPERIMENTAL WORK

A. Phase I - Fabrication Studies

1. PPQ Fabrication Problems

The PPQ resin is a solid high-molecular weight resin. Its chemical structural formula is shown in Figure 1. In order to evenly impregnate fiber with a solid resin, it is necessary to dissolve the resin in a solvent. PPQ has such a high-molecular weight that only dilute solutions will have low enough viscosities to flow well into the fiber bundles. Usually about a 10% solution of resin in solvent is required. This presents a problem in getting enough resin onto the fiber. If a tow of fiber is drawn through a bath of this 10% solution, it will not pick up enough resin even though it is dripping with solution. Some extra procedure is therefore required to get enough resin on the fiber.

Once the fiber has been impregnated with the resin solution, most of the solvent must be removed. How much solvent to remove becomes a problem. If too much solvent is removed before the final molding, the resin will not flow properly resulting in poor bonding and gaps or voids not filled by the resin. If, on the other hand, not enough solvent is removed before molding, some of the solvent may become trapped inside the laminate causing voids and thus lowering the strength. Meta-cresol, which has been found to be the best solvent for the PPQ resins, has a boiling point of 201°C. This means that considerable heating is required to remove the solvent. Furthermore, once the solvent content becomes less than about ¼ of the total prepreg weight, the impregnated strands become stiff and inflexible. This means that the strands must be held in the desired final position during drying.

Finally, PPQ requires a higher molding temperature than most resins, about 400°C (750°F).[1]

There were three basic problem areas, then, which required work:

(1) Fiber Impregnation with the PPQ Resin

[1]When conversion from degrees Fahrenheit to degrees Centigrade is within 1 C of a round number, the number is rounded off for ease of reading.

(2) Solvent Removal

(3) Panel Molding

2. Fiber Impregnation

Three basic methods of impregnating the carbon fiber with PPQ resin were investigated. The first involved winding continuous carbon fiber under slight tension around a 25.4 cm (10 inch) diameter metal drum which had been covered with a thin teflon sheet. The resin solution was poured over this fiber until the fiber was completely soaked and could hold no more solution. Then the drum was placed in an oven and some of the solvent dried out. After this drying, more resin solution was soaked into the fiber. Three such impregnations of the fiber with the solution were required in order to get enough resin on the fiber. The fiber was cut off the drum while still tacky and layed out flat on the teflon sheet backing. Further oven drying was then required.

The second method of impregnating the fiber consisted of placing the fibers by hand parallel to each other on teflon sheet backed with a sheet of heavy aluminum foil as shown in Figure 3. A pan was then formed around the fiber with the aluminum foil. The entire desired amount of resin solution was poured into the pan, and the solvent removed by slowly drying in the oven.

The third method ("frame method") consisted of winding the fiber under tension around an aluminum frame as shown in Figure 4. A measured amount of resin solution was poured into an aluminum foil pan with teflon lining and the frame was lowered into the pan.

3. Solvent Removal

The problem of solvent removal included consideration of how to get the solvent out of the impregnated fiber, how much to remove, and if heat drying was involved, what times and temperatures to use.

Three methods of solvent removal were studied: methanol precipitation, oven drying, and vacuum drying. The first method consisted of pouring methanol (methyl alcohol) over fibers which were soaking in a measured amount of resin solution. The methanol caused the solid resin powder to precipitate out of the solvent solution. After agitating the methanol for a time, it was poured off taking some of the solvent with it. The amount of solvent removed depended on how long the solution had soaked in the methanol.

The other two solvent removal methods involved heating the impregnated fiber either in an oven or in a vacuum. The solvent was thus removed by evaporation. Various heating times and temperatures were investigated.

4. Panel Molding

The study of molding conditions involved consideration of such parameters as molding temperature, pressure, and time-temperature rise rate; if and when breathing of the press (releasing the pressure) was required; and postcuring times and temperatures. For the basic PPQ resin, PPQ-1 (Figure 1), the following molding conditions were tried: Molding temperatures of 368°C (695°F) and 400°C (750°F), pressures of 200, 400 and 800 psi, molding times of one hour and 3½ hours, rise times to molding temperature from 15 minutes to 3½ hours, and postcure temperatures of 400°C (750°F) and 454°C (850°F). Because of the limited time available, these conditions were often varied several at a time and simultaneously with variations in solvent removal conditions.

5. Test Specimens

All the specimens prepared while investigating solvent removal and molding conditions (Phase I) were composite bars 15.2 cm x 0.635 cm x thickness (6" x 0.25" x thickness) where the thickness was usually about 0.25 cm. Later work involved making a few small panels (15.2 cm x 7.6 cm x thickness) in preparation for producing a large number of specimens for heat aging tests (Phase II). Courtaulds HMS fiber was used throughout the program because of its superior thermal stability. Impregnation of the fiber was accomplished using the third method ("frame method") described in section 2 above and 10% solutions of the PPQ-1 resin.

6. Test Methods

a. Solvent Content. The weight percent solvent contained in the prepregged strands just before molding was determined by measuring percent weight lost by the specimen during molding. (The molding temperatures used where 140° to 200°C above the boiling point of the solvent.) No resin is lost during molding since with PPQ there is very little flow. Any flow that takes place is due to the plasticizing effect of the solvent. It is hard enough to get the resin to flow out to the edge of the piece being laminated. Thus, any weight loss that takes place is due to loss of solvent. This is not an extremely accurate method since some solvent could remain trapped in the laminate. However, it was convenient and sufficiently accurate for our purpose of obtaining a relative measure of the amount of solvent left in the prepreg.

b. Flexural and Shear Strengths. All shear tests in this program were short beam interlaminar shear (ASTM D 2344-67), run at a span-to-depth ratio of 5. Flexural tests were 3 point bending according to ASTM D 790-66.

c. Fiber and Void Content. The volume fraction of fiber in the composite was calculated using equation (1) and the void content using equation (2) given below:

$$V_F = \frac{W_F \rho_C}{\rho_F} \qquad (1)$$

$$\% \text{ voids} = 100 \times \left[\frac{V_F (\rho_F - \rho_R)}{\rho_R} - \frac{(\rho_C - \rho_R)}{\rho_R} \right] \quad (2)$$

where V_F = volume fraction of fiber
W_F = weight fraction of fiber
ρ_C = density of the composite
ρ_F = density of the fiber
ρ_R = density of the resin

The weight fraction of fiber, W_F, was determined by measuring the total weight of the laminate and knowing the amount of fiber contained therein. The accuracy of this method depends mainly upon the uniformity in weight per unit length of the fiber. The greatest uncertainty and hence source of error in the void content calculations, however, is probably in the value of fiber density. As long as the same value of fiber density is used in all the void calculations being compared, the relative relationships will be about the same even if the absolute values are somewhat in error.

d. Torsional Braid Analysis. Torsional Braid Analysis (TBA) is a dynamic mechanical test method (references (16) and (17)). It is similar to the torsional pendulum in that it provides a measure of the damping and rigidity of the test specimen versus temperature. The TBA specimen consists of the polymer to be tested on a multifilamented glass fiber substrate (or braid). The changes observed in mechanical properties are attributed to changes in the polymer alone. This differs from the torsional pendulum where the properties measured are of the entire composite.

B. Phase II - Heat Aging

1. Fabrication of Specimens

Flexural specimens, 6.35 cm x 0.635 cm x ~0.3 cm (2.5" x 0.25" x ~ 0.1"), and shear specimens, ~1.5 cm x 0.635 cm x ~0.3 cm, for heat aging studies were cut from unidirectional panels, 15.2 cm x 7.6 cm x ~0.3 cm (6" x 3" x ~0.1"). Two sets of panels were made: the first set (6 panels) using a 10% solution of PPQ-1 in m-cresol (inherent viscosity of resin = 0.75) and the second set (4 panels) using a 5% solution of PPQ-1 in m-cresol (inherent viscosity of resin = 2.0). The second batch of resin was of higher purity and hence higher molecular weight and inherent viscosity than the first. For this reason, the more dilute solution of 5% was required to get good handling properties.

The fabrication procedure for these panels was as follows:

a. Wind Frame. Courtaulds HMS fiber was wound around an aluminum frame (15.2 cm x 25.4 cm inside dimensions) as shown in Figure 4. The fiber spacing was 4.7 strands/cm (12 strands/inch).

b. Dip in Dilute Solution. The frame was then dipped in a ½% solution of the resin to get good wetting. The soaked frame was allowed to stand 15 minutes on a side so that the excess solution would drip off.

c. Dry. Some of the solvent was dried off by rotating the frame for one hour in a 93°C (200°F) draft oven.

d. Main Impregnation. A measured amount of the 10% (or 5%) resin solution was spred evenly in an aluminum foil pan with teflon sheet lining (Figure 5). The frame was then lowered into the pan and the solution allowed to soak up through the fiber. The pan and frame assembly was placed in a 93°C (200°F) draft oven for one hour (about three hours for the 5% solution) then the frame was flipped over and the other side placed in a similar pan of resin for one hour (about three hours for the 5% solution). The frame was taken out of the pan and dried 1½ hours (at least two hours for the 5% solution) more at 93°C (200°F). At this point, the strands on the first side of the frame were cut off the frame and placed on a second frame. By now the prepregged sheets were stiff, but some wet sections often remained on the surfaces which were enclosed on the inside of the frame. Further drying (about one hour) was done at 93°C until the sheets appeared completely dry and stiff. This was followed by a final drying of three hours at 150°C (300°F). The prepregged sheets were then removed from the frames and cut into six 15.2 cm x 7.6 cm (6" x 3") sheets. The solvent content of the prepreg so prepared ran from 6 to 7% of the total weight.

e. Molding. The six 15.2 cm x 7.6 cm prepregged sheets were stacked on top of one another and placed in a 7.6 cm (3 inch) wide open ended mold. The mold was put in a hot 232°C (450°F) press. A 400 psi pressure was applied during a mold temperature rise of 216°C to 260°C (420°F to 500°F) and maintained until about 343°C (650°F) was reached. At this point the pressure was released to allow the remaining solvent to escape. Pressure was reapplied, released once more, and reapplied. Temperature was taken up to 400°C (750°F). This entire procedure took 26 minutes. After this the 400°C temperature and 400 psi pressure were maintained for one hour. The mold was then cooled slowly under pressure.

f. Postcures. A further curing or a "post curing" of the composite panels was required to crosslink the PPQ resin and thus obtain better mechanical properties at high temperatures. Half of the first set of panels were postcured for 20 hours at 454°C (850°F). The other half of the first set and all of the second set were postcured for 20 hours at 400°C (750°F). Panels were clamped between steel plates during postcure in order to keep them flat. All postcuring was done in a nitrogen atmosphere.

2. Heat Aging Tests

The fabricated panels were cut up into flexural specimens and short beam shear specimens. A set of "control" flexural and shear specimens from each set of panels were tested for room temperature strength properties. The remainder of the specimens were divided into groups of six to eight specimens and placed in a circulating air oven for long-term high-temperature heat aging. Heat aging was run at 370°C ± 5°C (700°F ± 10°F) for the first set of panels and at 343°C ± 5°C (650°F ± 10°F) for the second set of panels. After a given length of time at the heat aging temperature, specimens were removed from the oven and tested at that same temperature for strength retention. Percent weight loss was also measured.

Heat aging tests were also run on some of the solid resin powder measuring percent weight loss versus time.

RESULTS AND DISCUSSION

A. Phase I - Fabrication Studies

1. Impregnation of the Fiber

Table 1 outlines the advantages and disadvantages of the three methods of impregnating the fiber. The first method of winding the fiber around a drum ("drum method") had an advantage of being a quick easy way to lay up the strands of fiber. It also kept the strands straight during impregnation. The problem with this method was that the strands had to be impregnated several times in order to get the resin content high enough. The second method of laying the fiber up by hand in a pan ("pan method") required only a single impregnation, but it was time consuming to lay up the fibers and also difficult to keep them straight. The third method of winding the fiber around a frame and dipping this into a pan containing the resin solution ("frame method") was designed to eliminate the problems with the first two methods. The frame method became the standard method, and was used for the majority of the work.

2. Solvent Removal and Molding Studies

Early solvent removal studies were conducted using a PPQ resin containing a sulfone group. The structural formula is shown in Figure 2. The high temperature properties of this resin are not as good as those of the PPQ-1 (Figure 1). However, the handling problems were about the same since the resins were very similar and the same solvent (m-cresol) was used with both. Also the sulfone containing resin was available before the PPQ-1. Various conditions of oven and vacuum drying were investigated. The results are summarized in Table 2. Drying temperatures were varied from 93°C (200°F) to 177°C (350°F) in the vacuum and from 120°C (250°F) to 204°C (400°F) in the oven. Resulting shear strengths varied from 6.3 x 10^6 n/m^2 (910 psi) to 43.9 x 10^6 n/m^2 (6370 psi) and seemed to

depend more on the void content of the molded bar than on anything else. There was no advantage to the vacuum drying. Long drying times (6 to 9 hours) in the oven at 120°C (250°F) and 150°C (300°F) resulting in 7 to 8% volatiles in the prepreg gave the lowest composite void contents and highest shear strengths.

Because of the limited time available, solvent removal and molding conditions were varied simultaneously in studies with the PPQ-1 resin. Table 3 summarizes the various conditions used along with the resulting solvent contents, void contents, and shear strengths. Void contents ranged from 4% to 14% while short beam shear strengths ranged from 24.2 x $10^6 n/m^2$ (3510 psi) to 43.3 x $10^6 n/m^2$ (6290 psi). It was found that the method of removing solvent with methanol gave poor results. Vacuum drying, as shown earlier, was also of no advantage. Most of the solvent removal was effected by oven drying using temperatures in the 93°C to 150°C range.

A molding pressure of 400 psi was found to give good results and was used for most of the laminated bars. An increase of pressure to 800 psi on Bar N22 gave no improvement in shear strength over the bars molded at 400 psi.

A molding temperature of 400°C (750°F) was used with the PPQ-1 resin so as to be safely above the glass transition temperature, the onset of which was reported as 317°C (reference (14)). A Torsional Braid Analysis (TBA) was run on the PPQ-1 resin. The resulting plots of mechanical damping index and relative rigidity versus temperature are given in Figures 6 and 7. The peak of the damping curve (indicating glass transition) occurred around 365°C (690°F). Bar N29 (see Table 3) was molded quite satisfactorily at just a few degrees above this temperature. Thus any molding temperature somewhat above the glass temperature would probably be satisfactory.

The void contents obtained seemed to depend largely on the amount of solvent in the prepreg before molding. Figure 8 shows a plot of void content vs. solvent content values taken from Table 3. The data are somewhat scattered since there are other parameters involved, but the trend of higher void contents accompanying higher solvent contents is clear. A corresponding decrease in shear strength with increasing solvent content is shown in Figure 9. Unfortunately, the lower the solvent content, the harder it is to get flow during molding. Many of our bars did not show good flow all the way out to the ends because of the low solvent content in the prepreg. Often only half of the length of the bar was actually molded well enough to be worth testing. Thus, it is desirable to leave some solvent in the prepreg to plasticize the resin and provide flow during molding. Furthermore, removal of solvent to less than 5% would require long drying times (more than 6 hours for a 10% solvent solution).

An additional set of test bars (not listed in Table 3) was made by first dipping the fiber in a dilute (1/2%) solution of PPQ, drying slightly, then doing the main impregnation with the 10% solution. The same technique was tried on some 7.6 cm x 15.2 cm (3" x 6") panels. It was found that use of dilute solution gave better flow (out to the ends of the bar or panel). Strength values were about the same as those obtained without using the dilute solution.

Breathing of the press, that is, releasing the pressure during the initial heating stage, was found to aid greatly in releasing trapped solvent providing the breathing was done around 343°C (650°F). During fabrication of Phase II panels, breathing at around 343°C (650°F) was found more successful than at the lower temperatures (170°C - 252°C) tried earlier (Table 3). In fact breathing at these lower temperatures was so ineffective that there was no noticeable difference in the resulting composite strengths between breathing or not breathing in the Phase I work. All of the panels made for heat aging studies in Phase II were made by breathing at the 343°C (650°F) temperature.

B. Phase II - Heat Aging Tests

1. Room Temperature Properties

Measurements of fiber content, density, and void content were made on each of the composite panels fabricated for heat aging studies. These values are listed in Table 4 along with postcure conditions. A group of control specimens (containing flexural and shear specimens from each panel in the set) was tested to determine the room temperature strengths. These results are listed in Table 5.

For the first set of panels (made with the 10% PPQ solution of inherent viscosity = 0.75) the fiber content was 60% $\pm$ 1% by weight and the void content ranged from 2 to 4%. Room temperature strengths depended on the postcure temperature. Those panels postcured at 400°C (750°F) had an average shear strength of 50.1 x $10^6 n/m^2$ (7260 psi) and an average flexural strength of 8.14 x $10^8 n/m^2$ (118,000 psi). These shear and flexural strengths are roughly 80% of strengths obtained with Courtauld's HMS fiber/epoxy resin composites (reference (18)). Exact comparisons are not possible because of different fiber contents, fiber lots, and void contents.

The panels postcured at 454°C (850°F) showed lower average strengths of 43.9 x $10^6 n/m^2$ (6370 psi) in shear and of 6.14 x $10^8 n/m^2$ (89,100 psi) in flexure. The large variation in flexural strength values is due to the fact that some of the specimens failed at low values in shear while others failed in flexure at values equivalent to those of the 400°C postcured group. The generally lower strengths of the 454°C postcured group indicates that this postcure (20 hours at 454°C in Nitrogen) somewhat degraded the resin.

The second set of panels (made with the 5% PPQ solution of inherent viscosity = 2.0) had fiber contents of 63% $\pm$ 1% by weight and void contents of 7%. This higher void content was due to the increased difficulty in using the 5% solution and the fact that there was not enough time available to learn how to compensate for the additional problems. The main effect of this higher void content was to lower the shear strength. The average room temperature shear strength was 33.6 x $10^6 n/m^2$ (4880 psi).

Numerous shear failure were encountered during flexural tests causing considerable scatter in failure values. The average flexural strength was 7.45 x $10^8 n/m^2$ (108,000 psi).

2. Heat Aging Results

a. First Set of Panels. The flexural and shear specimens from the first set of panels were divided into groups and put into a 371°C (700°F) circulating air oven for heat aging. After 192 hours the first group was removed for strength tests. The specimens were found to have already lost a considerable amount of weight. So much of the resin had burned off that the planned strength tests were not run and the test was terminated. For those specimens postcured at 400°C (750°F), the average weight loss was 20% for the flexural specimens and 22% for the shear specimens. The specimens which had been postcured at 454°C (850°F) lost even more weight. Mostly fibers were left so that the individual specimens were not distinguishable.

There were two reasons for this disastrous result. First, the actual oven temperature during the last 100 hours averaged more like 377°C (710°F) than 371°C (700°F) and may occasionally have been as high as 379°C (715°F). Secondly, the purity and molecular weight of the batch of resin used was not sufficiently high as indicated by the low inherent viscosity (0.75) of the resin. The real importance of the latter was not recognized until further heat aging was done.

b. Weight Loss of Resin Powders and of Composites. Heat aging tests to determine percent weight loss vs. time were conducted on the solid resin powder and on pieces of the composite control specimens from the first set of panels. Three batches of resin with different inherent visocities were tested. None of these batches was the same as was used in fabricating the composite panels. The results are shown in Figures 10 and 11. Comparing these two figures, it is clear that the resin powders lose weight much faster than the resin in a cured composite. This is due to the larger exposed surface area of the powder. There was considerable difference between the percent weight lost by the resin powder at 343°C (650°F) and that lost at 371°C (700°F). Batch 1 (see Figure 10), for example, lost 11% after 157 hours at 343°C as compared with 39% after 150 hours at 371°C. The higher weight loss of resins with lower inherent viscosities was noticeable for heat aging run at 343°C. The rate of weight loss at 371°C was too high and the difference in inherent viscosities of the two resin batches too

small to show any difference in their thermal stabilities at that temperature. These low inherent viscosity resins lost over 70% of their weight after 200 hours at 371°C. Clearly these resin batches were nowhere near the quality of the original PPQ (reference (14)). Apparently the properties of the PPQ resin degrade rapidly with increased impurities in the resin and/or off-stoichiometric mixtures of the resin.

The composite heat aging curves (Figure 11) show a rapid weight loss of those composites samples postcured at 454°C (850°F), indicating that the postcure at this temperature has degraded the resin. The weight loss on the 400°C postcured samples after heat aging for 195 hours at 371°C (700°F) was 12%. This was less than the 20 to 22% lost during the less carefully controlled heat aging on the first set of panels reported in section 2a above. It verifies the fact that the higher oven temperature was partly responsible for the excessive composite weight loss in the first test. Twelve percent weight loss is still quite high, however. This is attributed to impurites in the resin.

c. Second Set of Heat Aging Panels. From the first set of panels it was learned that a higher purity, higher molecular weight PPQ resin should be used to get the desired thermal stability. A second set of panels was made with a 5% solution of higher molecular weight resin (Inherent viscosity = 2.0). These were all postcured at 400°C since the higher 454°C postcure was found to degrade the resin causing more rapid weight loss during heat aging (Figure 11). Unfortunately, if the lower postcure temperature is used, there is a problem with thermoplastic failure of the composite under load at 371°C (700°F) and above (reference (15)). Because of this and because of the high weight loss of the lower inherent viscosity resins at 371°C (Figure 10), a heat aging temperature of 343°C (650°F) was chosen for this second set of specimens.

Flexural and shear strengths were measured at 343°C (650°F) after one hour, 168 hours, 264 hours, and 360 hours of heat aging at 343°C. The results of these tests are listed in Tables 6 and 7. Comparing the values measured at 343°C after one hour at temperature with those measured at room temperature (Tables 5), it is evident that there is no initial loss of strength properties at 343°C over room temperature strengths. The initial failures of the shear specimens at this temperature were thermoplastic. Most of the flexural specimens exhibited true flexural failures, with the fibers breaking, and only a few specimens failed improperly by shearing the resin.

There was considerable scatter in all the strength tests conducted on this set of specimens. Individual failure points are plotted in Figures 12 and 13. Despite the scatter, the results of the heat aging test are quite clear. Based on average values and the initial strength at temperature, the strength loss after 168 hours at 343°C (650°F) is 68% for flexural specimens and 61% for shear specimens. Cracking and delamination of the specimens especially the flexural specimens, was evident after removal from

the oven and before the strength tests were run. This condition worsened with longer heat aging exposure times. The measured average strengths also continued to decrease with longer exposure. All of the flexural specimens after 168 hours or more of heat aging failed in shear during the strength tests. Three of the twelve shear specimens which were heat aged for 360 hours were split completely in two after that exposure.

The percent weight loss due to heat aging was measured on the composite specimens and on resin powder from the batch of resin used in fabricating the composites. The weight loss results are plotted in Figure 14. An average of 3% weight had been lost by the composite specimens after 168 hours at 343°C (650°F). While this value is reasonably low, the corresponding strength loss after 168 hours is quite high. This apparent discrepancy between weight and strength loss would indicate that while the resin has not significantly oxidized or burned away after 168 hours, it has degraded or at least the resin-fiber bond had degraded. The degradation at this point was sufficient to cause loss of more than half of the mechanical strength.

SUMMARY AND CONCLUSIONS

As a result of this program, successful methods of fabricating flat carbon composites using PPQ (phenylated polyquinoxaline) resin have been developed. Carbon/PPQ composites have been fabricated with room temperature shear and flexural strengths about 80% of those obtained with graphite/epoxy composites and void contents of 2 to 4%.

Above 343°C (650°F) the composites were found to fail thermoplastically under flexural loading when postcured at 400°C (750°F). Postcure at 454°C (850°F) eliminated the problem of thermoplastic failure but significantly degraded the resin.

Measurements of composite shear and flexural strength at 343°C (650°F) indicate no initial loss of strength at this temperature over room temperature properties. After 168 hours at 343°C, however, the resin matrix had degraded sufficiently to cause loss of more than half of the composite shear strength and flexural strength. Thus, the Carbon/PPQ composites cannot be recommended for long-term high temperature use above 600°F although they may be quite useful in short-term (less than 24 hours) applications.

RECOMMENDATIONS

1. Additional measurements of strength versus heat aging time would be useful to determine where between one hour and 168 hours the resin degrades at 343°C (650°F).

2. Work to develop chemical crosslinking of the resin is needed (and now being conducted at Whittaker Corporation) to eliminate the thermoplastic failure of the composites at 400°C and above without the degrading 454°C postcure.

3. Lower temperature heat aging tests are needed to determine at what temperature the resin is useful at 1,000 hours, or 10,000 hours.

4. In the area of fabrication, considerable work is needed before low-void, high-strength-laminates in curved shapes can be made.

ACKNOWLEDGEMENTS

The assistance of Mr. Richard F. Jones and Mr. Walter Johnson, who performed the actual fabrication of the PPQ composites, is gratefully acknowledged.

REFERENCES

1. Mantell, C. L., Carbon and Graphite Handbook, John Wiley and Sons, Inc., New York, 1968, pp. 323-377.

2. J. H. Freeman et al., AFML-TR-65-188, "Development of Polimide Resin Based Structural Adhesives and Laminates for Long Time High Temperature Use," Westinghouse Research Laboratories, Aug 1965.

3. J. K. Fincke et al., AFML-TR-67-136, "Synthesis of New Thermally Stable Polyimide Laminating Resins," Monsanto Research Corp., Aug 1967.

4. Wintermute, G., AFML-TR-67-273, "Fabrication and Test of High Temperature Honeycomb Core Sandwich Construction," Goodyear Aerospace Corp., Sep 1967.

5. Browning, C. E. and Marshall, J. A., AFML-TR-69-130, "Fabrication and Evaluation of Graphite Fiber Reinforced Polyimide Composites," Air Force Materials Laboratory, Nov 1969.

6. Rogers, K. F. and Kingston-Lee, D. M. "R. A. E. Polyimide Resin/Carbon Fiber Composites: Part 2-The Manufacture and Evaluation of Laminates," Royal Aircraft Establishment Technical Report 70049, Mar 1970.

7. Browning, C. E. and Marshall, J. A., "Graphite Fiber Reinforced Polyimide Composites," Journal of Composite Materials, 4, 390 (Jul 1970).

8. Hergenrother, P. M., Kerkmeyer, J. L., and Levine, H. H., "High-Temperature Structural Resins," Whittaker Corp. under Naval Weapons Contract NOW 64-0524-C, May 1965.

9. Hergenrother, P. M. and Levine, H. H., "High-Temperature Structural Resins," Whittaker Corp. under Naval Weapons Contract 66-0144-C, Jul 1966.

10. H. H. Levine et al., AFML-TR-67-429, "High-Temperature Resins for Structural Laminates and Adhesives," Whittaker Corp., Jan 1968.

11. Hergenrother, P. M. and Levine, H. H., "High-Temperature Structural Resins," Whittaker Corp. under Naval Ordnance Contract N60921-67-C-0221, Jun 1968.

12. Sanders, B. Y., Fitzgerald, W. P., and Levine, H. H., AFML-TR-69-303, "High-Temperature Resins for Structural Laminates and Adhesives," Whittaker Corp., Oct 1969.

13. Fitzgerald, W. P., Harrison, E. S., and Sanders, B. Y., AFML-TR-71-4, "High-Temperature Resins for Structural Laminates and Adhesives," Whittaker Corp., Feb 1971.

14. Augl, J. M. and Wrasidlo, W., NOLTR -69-120, "Synthesis and Evaluation of New Phenylated Polyquinozalines," Oct 1969.

15. Rafter, R. T. and Fitzgerald, W. P., "High-Temperature Polymer Development," Whittaker Corp. under Naval Ordnance Contract N60921-69-C-0073, Dec 1970.

16. Lewis, A. F. and Gillham, J. K., J. Appl. Polymer Science, 6, 422 (1962).

17. Gillham, J. K., in Thermoanalysis of Fibers and Fiber-Forming Polymers, Applied Polymer Symposia No. 2, (R. F. Schwenker, Jr., Ed.), Wiley-Interscience, N. Y. (1966) p. 45.

18. Simon, R. A., "Graphite Fiber Composites for Deep Submergence," Jan 1971, Not available.

TABLE 1

METHODS OF IMPREGNATING FIBERS

METHOD	ADVANTAGES	DISVANTAGES
DRUM	1. FIBERS MAINTAINED STRAIGHT 2. EASY TO SET UP	1. REQUIRES MULTIPLE IMPREGNATIONS
PAN	1. SINGLE IMPREGNATION REQUIRED	1. FIBERS BECOME WRINKLED 2. TIME CONSUMING SET UP
FRAME	1. FIBERS MAINTAINED STRAIGHT 2. SINGLE IMPREGNATION REQUIRED 3. LEAST TIME CONSUMING	

TABLE 2

Solvent Removal Study Using Sulfone-Containing PPQ Resin

Drying Condition: Initial +		% Solvent in Prepreg	Volume Fraction of fiber	Density (g/cm^3)	Void Content (%)	Interlaminar Shear Strength ($n/m^2 \times 10^{-6}$)	(psi)
OVEN	3 hrs at 120°C	19	0.38	1.38	11	26.8	3890
	9 hrs at 120°C	8	0.40	1.51	1½	41.4	6000
	2 hrs at 150°C	15	0.37	1.41	8	22.1	3210
	6 hrs at 150°C	7	0.36	1.49	1	43.9	6370
	2 hrs at 177°C	9	0.39	1.41	9	29.3	4250
	6 hrs at 177°C	3	0.35	1.42	6	26.5	3840
	1 hr at 204°C	8	0.36	1.41	7	19.9	2880
	3 hrs at 204°C	5	<0.24	<1	>34	6.3	920
VACUUM	2 hrs at 93°C	27	0.43	1.47	6½	26.8	3890
	6 hrs at 93°C	15	0.38	1.44	6	38.4	5570
	2 hrs at 120°C	28	0.39	1.38	11½	25.6	3710
	6 hrs at 120°C	14	0.35	1.45	4½	29.6	4290
	2 hrs at 150°C	7	0.35	1.44	5	21.8	3160
	6 hrs at 150°C	7	0.33	1.38	8	14.8	2100
	1½ hrs at 177°C	5	0.23	1.0	34	6.3	910
	4 hrs at 177°C	3	0.34	1.45	4	29.4	4260

NOTE: 1. Initial Drying Condition = 3 hrs. at 93°C for oven dry
= 1 hr. at room temperature + 3 hrs. at 66°C for vacuum dry.

2. Void Content calculations are based on a fiber density = 1.94 g/cm^3 (manufacturer's value)

3. Molding Condition: 1 hr. at 316°C under 400 psi pressure for all bars.

TABLE 3

Solvent Removal and Molding Studies on PPQ-1 Resin/Carbon Fiber Composites

Bar Number	Solvent Removal Condition	% Solvent in Prepreg	Molding Condition	Void Content (%)	Average Interlaminar Shear Strength $n/m^2 \times 10^{-6}$ (psi)
N10	OVEN 3 hrs at 93°C + 4 hrs at 150°C	5.6	In 400°C Press 1 hr/400°C/400 psi	4.6	42.9 (6220)
N11	METHANOL 6 hrs	19	In 400°C Press 1 hr/400°C/400 psi	---	24.2 (3510)
N12	METHANOL 13 hrs	6	In 400°C Press 1 hr/400°C/400 psi	---	31.2 (4520)
N13	METHANOL 28 hrs	2	In 400°C Press 1 hr/400°C/400 psi	---	32.1 (4660)
N14	VACCUM 3 hrs at 66°C + 2 hrs at 93°C	36	In 232°C Press Breathe at 232°C Up to 400°C at 55°/hr 1 hr/400°C/400 psi	10	36.1 (5230)
N15	VACUUM 3 hrs at 66°C + 12 hrs at 93°C	8.4	Same as N14	8.4	30.7 (4450)
N16	VACUUM 3 hrs at 66°C + 6 hrs at 93°C	17.8	Same as N14	9.6	30.1 (4370)
N17	OVEN 3 hrs at 93°C + 2 hrs at 120°C	10.9	In 232°C Press Breathe at 232°C Up to 400°C in ½ hr 1 hr/400°C/400 psi	10.1	38.4 (5570)
N18	OVEN 3 hrs at 93°C + 6 hrs at 120°C	7.5	Same as N17	9.1	38.3 (5550)
N19	OVEN 3 hrs at 93°C + 12 hrs at 120°C	6.9	Same as N17	9.7	38.7 (5620)

NOLTR 71-187

TABLE 3 (Cont.)

Bar Number	Solvent Removal Condition	% Solvent in Prepreg	Molding Condition	Void Content (%)	Average Interlaminar Shear Strength $n/m^2 \times 10^{-6}$ (psi)	
N20	OVEN 3 hrs at 93°C + 1 hr at 120°C	10.4	In 400°C Press Up to 400°C in 13 min 1 hr/400°C/400 psi	9.0	37.1	(5380)
N21	Same at N20	10.0	In 274°C Press Breathe at 170°C Up to 400°C in 70 min 3½ hrs/400°C/200 psi	8.8	33.5	(4860)
N22	Same at N20	13.2	In 400°C Press Breathe at 204°C 1 hr/400°C/800 psi	7.3	37.4	(5430)
N23	OVEN 3 hrs at 93°C	11.4	In 232°C Press Breathe at 204°C 1 hr/400°C/400 psi	9.3	31.4	(4560)
N24	OVEN 3 hrs at 93°C + 3 hrs at 150°C	6.3	In 400°C Press Up to 400°C in 15 min 1 hr/400°C/400 psi	6.4	40.5	(5880)
N25	OVEN 2 hrs at 93°C	16.9	In 274°C Press Breathe at 204°C 1 hr/400°C/400 psi	14.4	26.5	(3840)
N26	Same as N25	20.1	Same as N25	13.3	28.3	(4100)
N28	OVEN 3 hrs at 93°C	11.2	In 218°C Press Breathe at 218°C Up to 400°C in 60 min 1 hr/400°C/400 psi	8.7	36.3	(5270)
N29	OVEN 3 hrs at 93°C + 2 hrs at 120°C	7.3	In 232°C Press Breathe at 252°C Up to 368°C in 40 min 1 hr/368°C/400 psi	7.0	43.4	(6290)
N30	OVEN 3 hrs at 93°C + 3 hrs at 150°C	5.4	In 400°C Press Up to 400°C in 22 min 1 hr/400°C/400 psi	4.3	42.5	(6160)

TABLE 3 (Cont.)

Bar Number	Solvent Removal Condition	% Solvent in Prepreg	Molding Condition	Void Content (%)	Average Interlaminar Shear Strength $n/m^2 x10^{-6}$ (psi)
N31	OVEN 3 hrs at 93°C + 3 hrs at 120°C	7.4	In 232°C Press Breathe at 227°C Up to 400°C in 50 min 1 hr/400°C/400 psi	6.2	---
N32	Same as N31	5.9	Same as N31	7.2	---
N33	Same as N31	7.5	Same as N31	6.3	41.9 (6070)
N34	OVEN 4 hrs at 93°C	9.1	Same as N31	6.3	---
N35	Same as N34	7.7	Same as N31	8.1	---
N36	Same as N34	8.1	Same as N31	7.2	---

NOLTR 71-187

TABLE 4
Properties of Carbon Fiber Composite Panels made for Heat Aging Tests

Panel Set (1)	Panel Number	Postcure (2)	% Weight Lost in Postcure	Fiber Content (Weight %)	Density (g/cm^3)	Void Content (%) (3)
1	9	20 hrs at 400°C	--	60	1.50	4
1	10	20 hrs at 400°C	--	59	1.49	4
1	11	20 hrs at 400°C	0.6	60	1.49	4
1	12	20 hrs at 454°C	2.3	60	1.52	2
1	13	20 hrs at 454°C	2.4	61	1.50	4
1	14	20 hrs at 454°C	2.3	60	1.51	3
2	18	20 hrs at 400°C	0.7	63	1.47	7
2	19	20 hrs at 400°C	0.8	62	1.465	7
2	20	20 hrs at 400°C	0.7	64	1.47	7
2	21	20 hrs at 400°C	0.6	63	1.47	7

(1) Panel Set #1 was made using a 10% solution of PPQ-1 resin (inherent viscosity = 0.75).

Panel Set #2 was made using 5% solution of PPQ-1 resin (inherent viscosity = 2.0).

All panels were cured for 1 hour at 400°C under 400 psi pressure.

(2) All postcures done in Nitrogen.

(3) Based on manufacturer's value of fiber density = 1.94 g/cm^3 (Courtaulds HMS).

TABLE 5
Room Temperature Flexural and Interlaminar Shear Strengths
of Carbon Fiber Composite Control Specimens

Property	Panel Set #1[1] 400°C Postcured Group	Panel Set #1[1] 454°C Postcured Group	Panel Set #2[2]
Avg. Flexural Strength, $10^8 n/m^2$ (ksi)	8.14 (118)	6.14 (89.1)	7.45 (108)
Coeff. of Variation (%)	6.6	35.3	42.4
No. of Specimens	7	5	7
Types of Failure	all Flex	2 Flex, 3 shear	2 Flex, 5 shear
Span/Depth Ratio[3]	22	19	18
Avg. Interlaminar Shear Strength, $10^6 n/m^2$ (ksi)	50.1 (7.26)	43.9 (6.37)	33.6 (4.88)
Coeff. of Variation (%)	17.1	15.4	10.5
No. of Specimens	7	6	6
Type of Failure	all shear	all shear	all shear

(1) Panel Set #1 was made using a 10% solution of PPQ-1 (Inherent viscosity = 0.75).

(2) Panel Set #2 was made using a 5% solution of PPQ-1 (Inherent viscosity = 2.0).

(3) The high temperature flexural fixture (also used in room temperature tests for comparison purposes) had a fixed span of 5.08cm. The resulting span/depth ratio varied depending on specimen thickness.

TABLE 6
Flexural Strength After Heat Aging of PPQ-Carbon Fiber Composites (Panel Set #2)

Test Condition (1)	Average (2) Strength $10^8 n/m^2$ (psi)	Range $10^8 n/m^2$ (psi)	Number of Specimens	Type of Failure
At 343°C after 1 hr at 343°C	7.79(113,000)	4.69-9.24(68,000-134,000)	8	5 flex, 3 shear
At 343°C after 168 hrs at 343°C	2.51(36,400)	1.53-3.22(22,200-46,700)	7	all (3) shear
At 343°C after 264 hrs at 343°C	2.07(30,100)	1.21-2.68(17,500-38,900)	7	all (3) shear
At 343°C after 360 hrs at 343°C	1.20(17,400)	0.52-2.10(7550-30,400)	7	all (3) shear
At 22°C after 360 hrs at 343°C	1.03(15,000)	0.51-1.95(7330-28,200)	7	all (3) shear

(1) Heat Aging was conducted in circulating air oven at average temperature of 343°C (650°F ± 10°F).

(2) Span/Depth Ratio = 18. (See Footnote (3), Table 5.)

(3) Cracking and delamination of all flexural specimens were evident after heat aging and before the strength test. Condition was worse the longer the specimens had been heat aged.

TABLE 7

Interlaminar Shear Strength After Heat Aging of PPQ-Carbon Fiber Composites (Panel Set #2)

Test Condition[1]	Average Shear Strength $10^6 n/m^2$ (psi)	Range $10^6 n/m^2$ (psi)	Number of Specimens	Type of Failure
At 343°C after 1 hr at 343°C	33.0 (4780)	25.1-38.3 (3640-5560)	5	thermo-plastic
At 343°C after 168 hrs at 343°C	12.9 (1870)	10.6-18.1 (1540-2630)	6	shear[4]
At 343°C after 264 hrs at 343°C	8.41 (1220)	5.10-13.6 (740-1970)	6	shear[4]
At 343°C after 360 hrs at 343°C	7.58 (1100)[2]	6.07-11.4 (880-1650)	4[2]	shear[4]
At 22°C after 360 hrs at 343°C	6.34 (920)[3]	4.27-8.55 (620-1240)	5[3]	shear[4]

(1) Heat Aging was conducted in circulating air oven at average temperature of 343°C (650°F ± 10°F).

(2) Two specimens included in the original group but not averaged here split completely into two pieces during heat aging.

(3) One specimen included in the original group but not averaged here split completely into two pieces during heat aging.

(4) All these specimens appeared to have true shear failures. No permanent deformation due to plastic failure was visible. Not all of the loading curves were linear, however. In this case, the point where the curve departs from straight line was chosen as failure point.

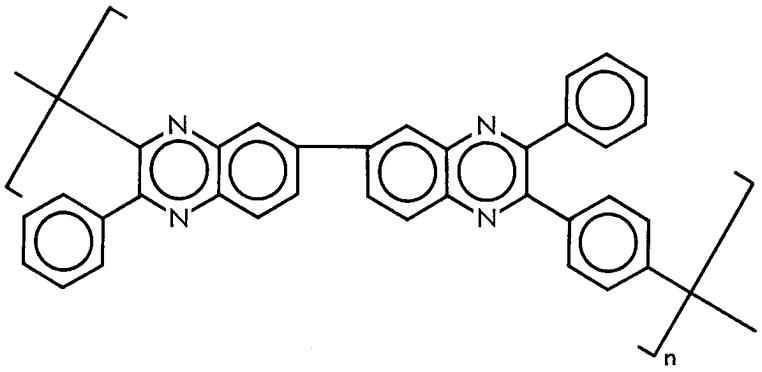

FIG. 1 STRUCTURAL FORMULA OF PHENYLATED POLYQUINOXALINE RESIN (PPQ-1)

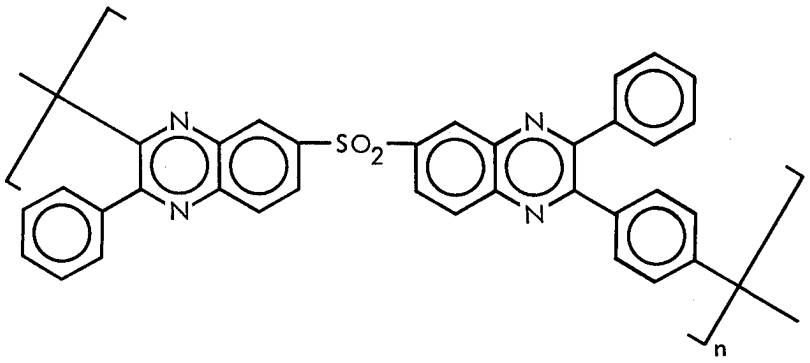

FIG. 2 STRUCTURAL FORMULA OF PPQ RESIN CONTAINING SULFONE

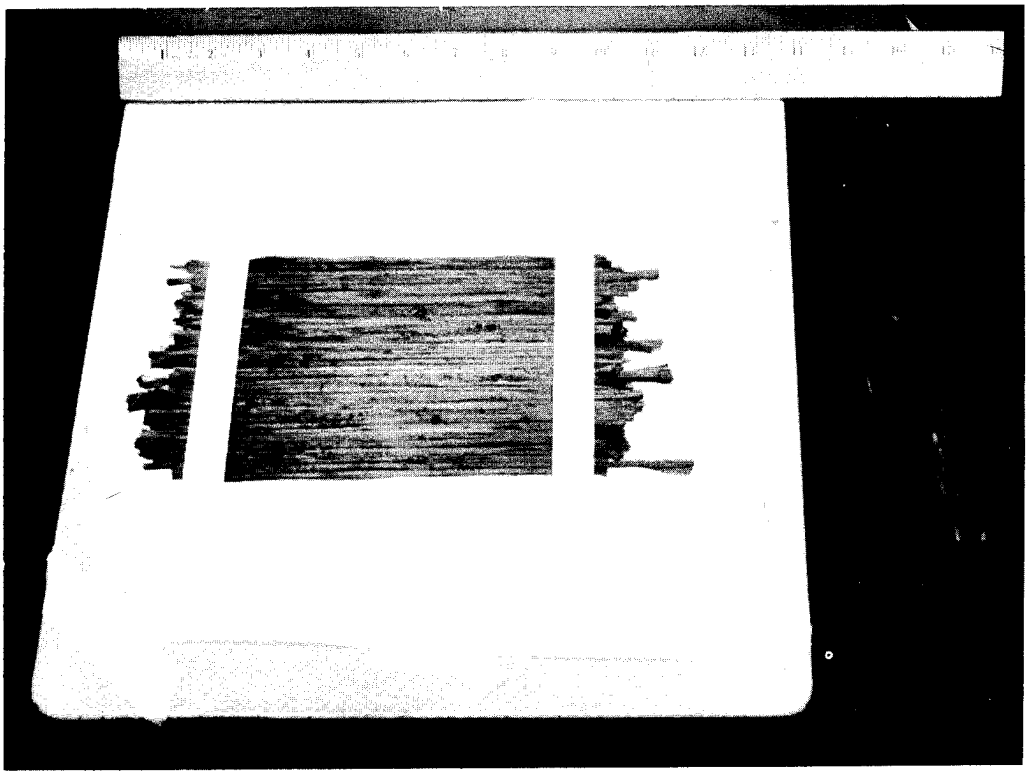

FIG. 3 PAN METHOD OF IMPREGNATING CARBON FIBERS WITH PPQ RESIN

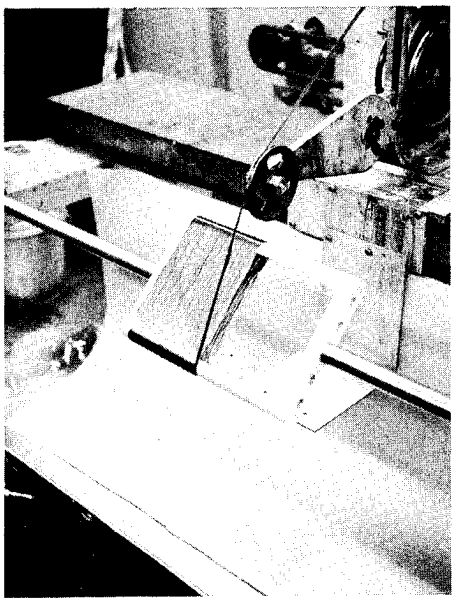

FIG. 4 WINDING FIBER AROUND ALUMINUM FRAME

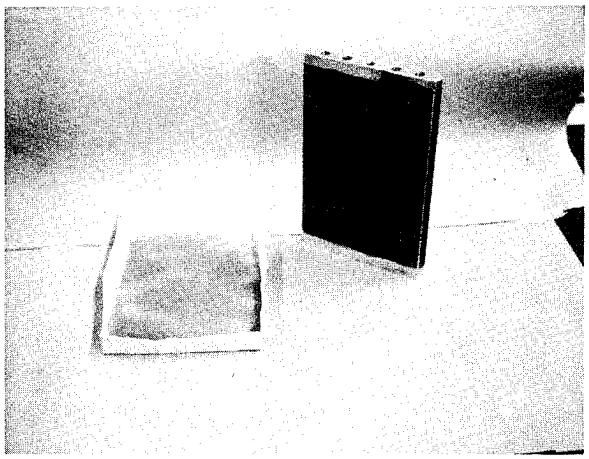

FIG. 5 PAN CONTAINING PPQ SOLUTION AND FRAME WITH FIBER

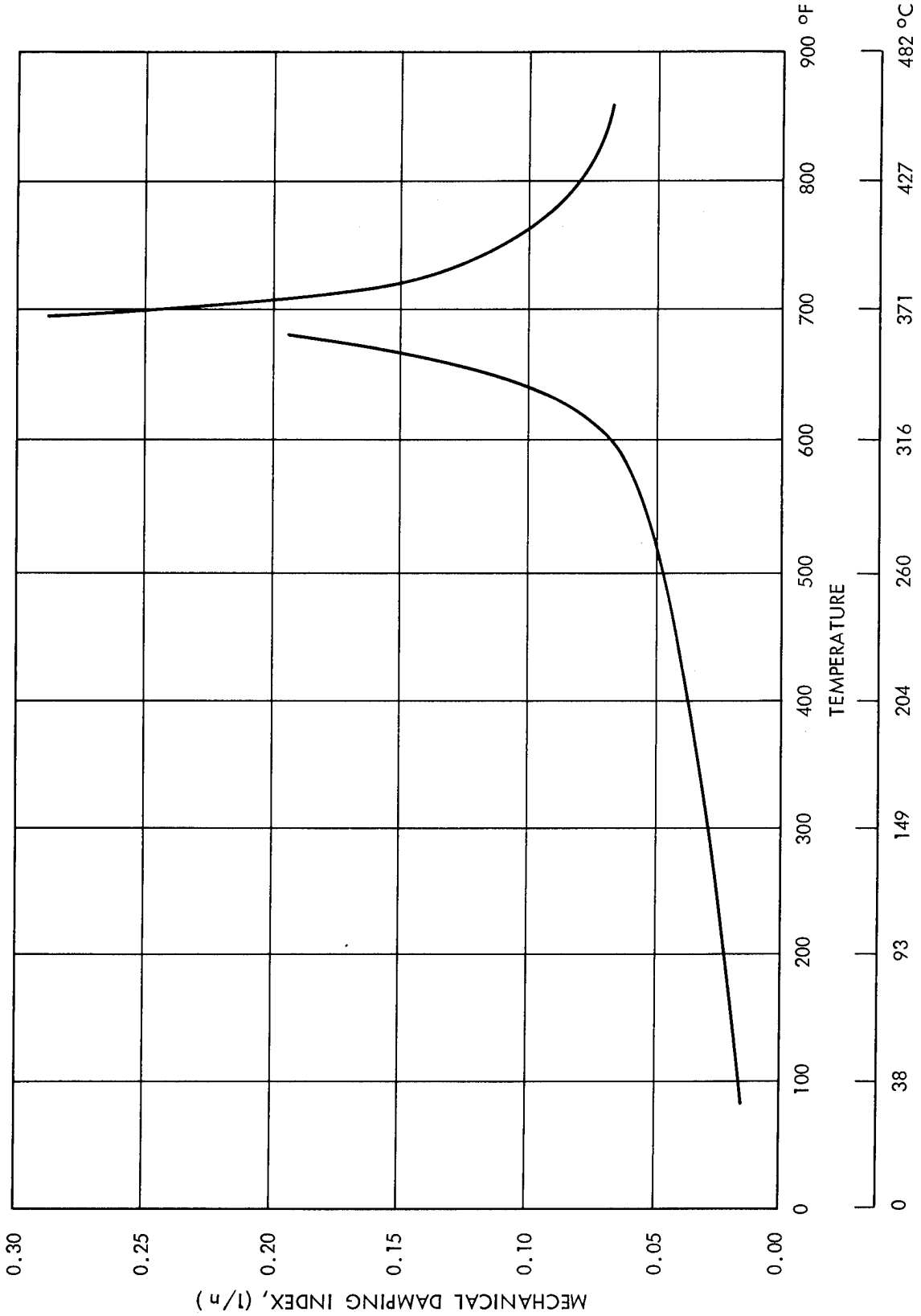

FIG. 6 TORSIONAL BRAID ANALYSIS OF PPQ-1 RESIN: MECHANICAL DAMPING INDEX VS. TEMPERATURE

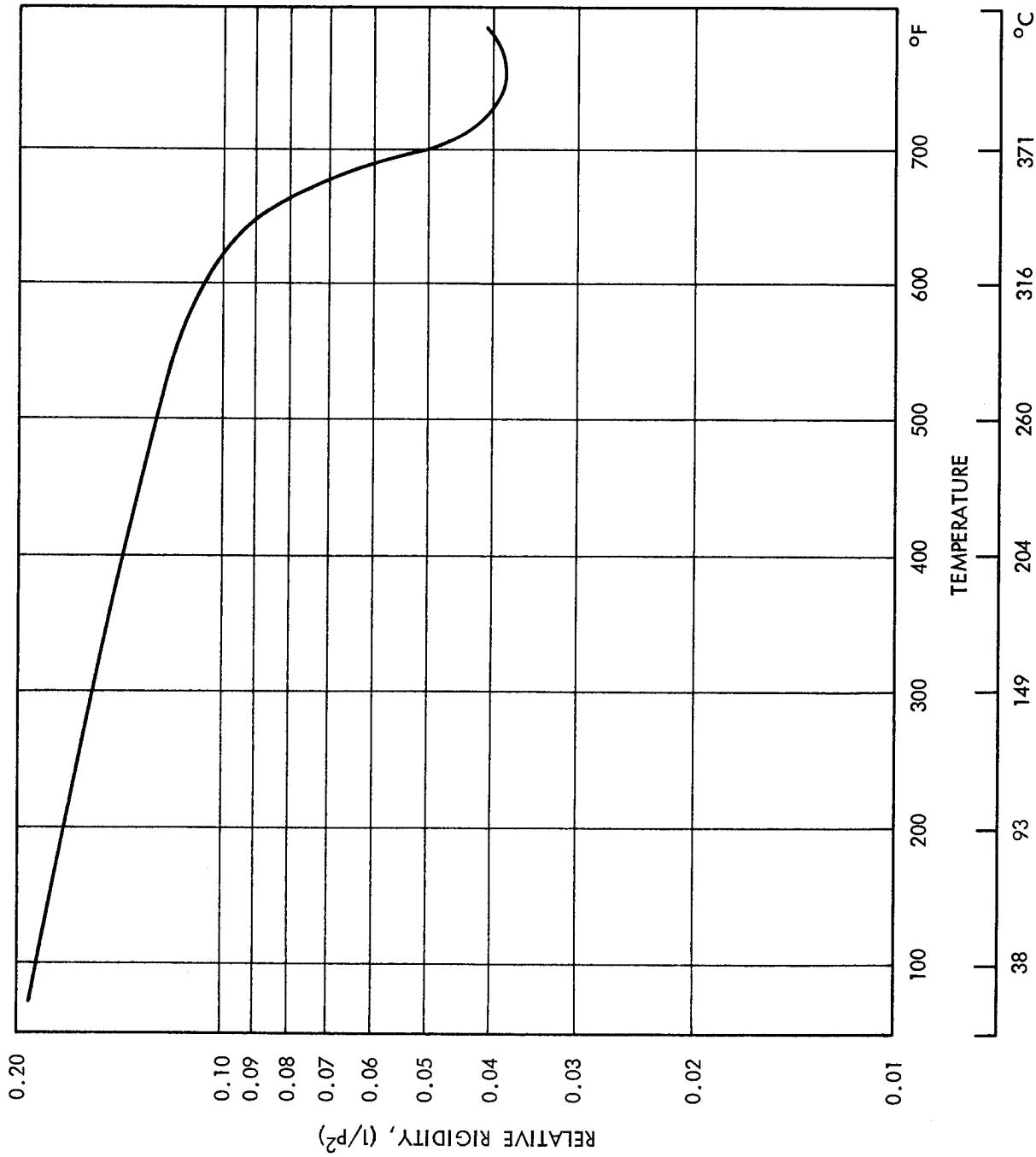

FIG. 7 TORSIONAL BRAID ANALYSIS OF PPQ-1 RESIN: RELATIVE RIGIDITY VS. TEMPERATURE

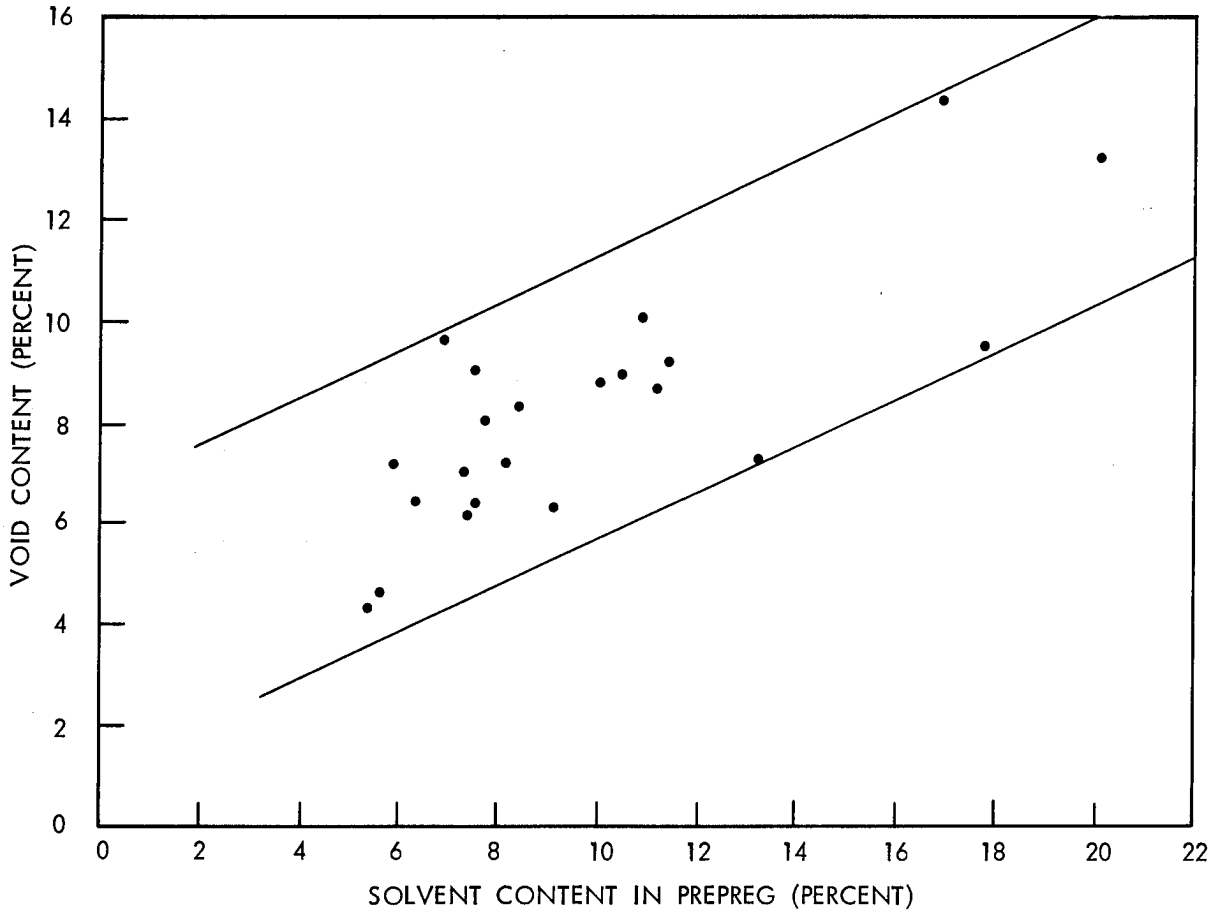

FIG. 8 COMPOSITE VOID CONTENT VS. PREPREG SOLVENT CONTENT

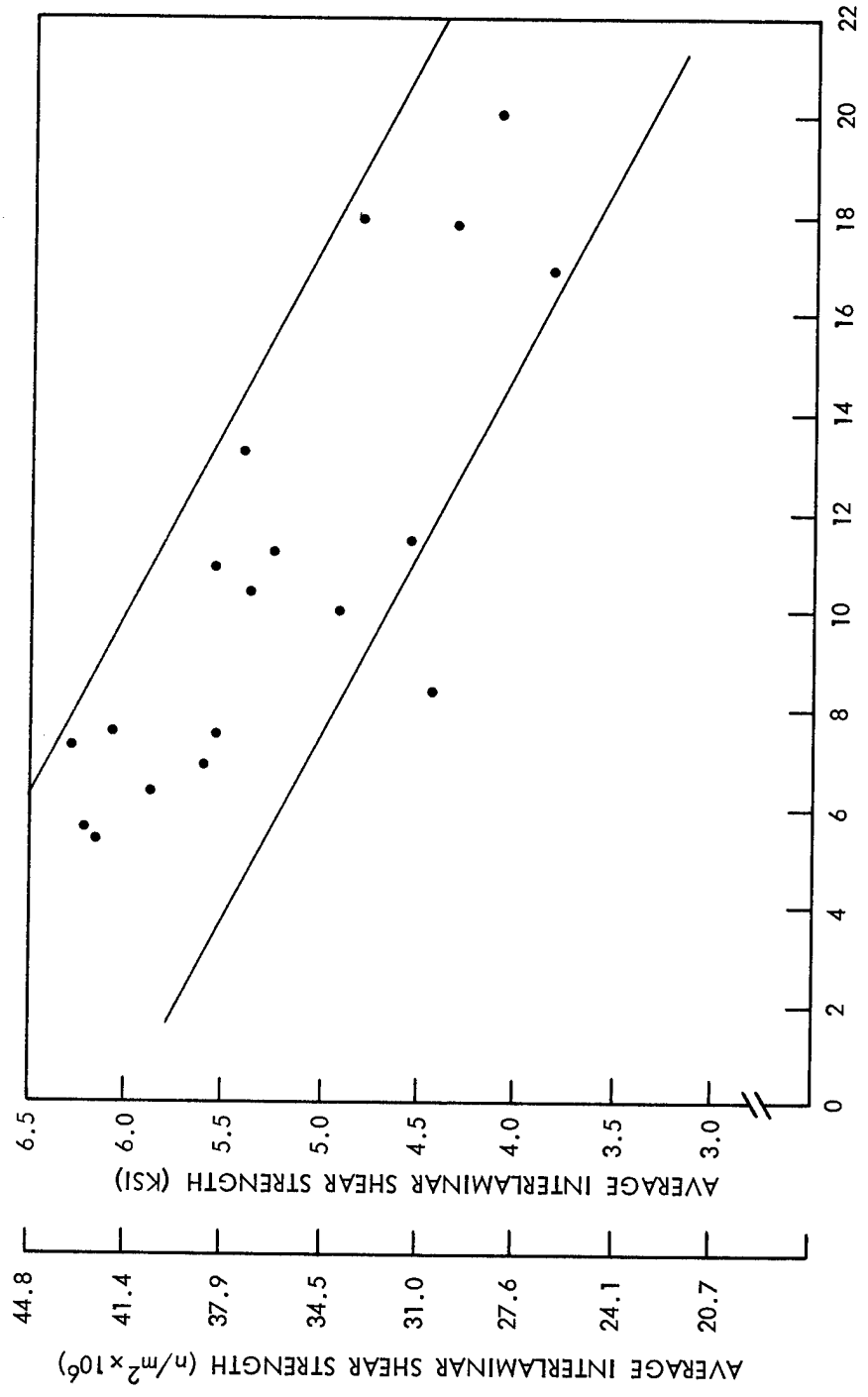

FIG. 9 COMPOSITE INTERLAMINAR SHEAR STRENGTH VS. PREPREG SOLVENT CONTENT

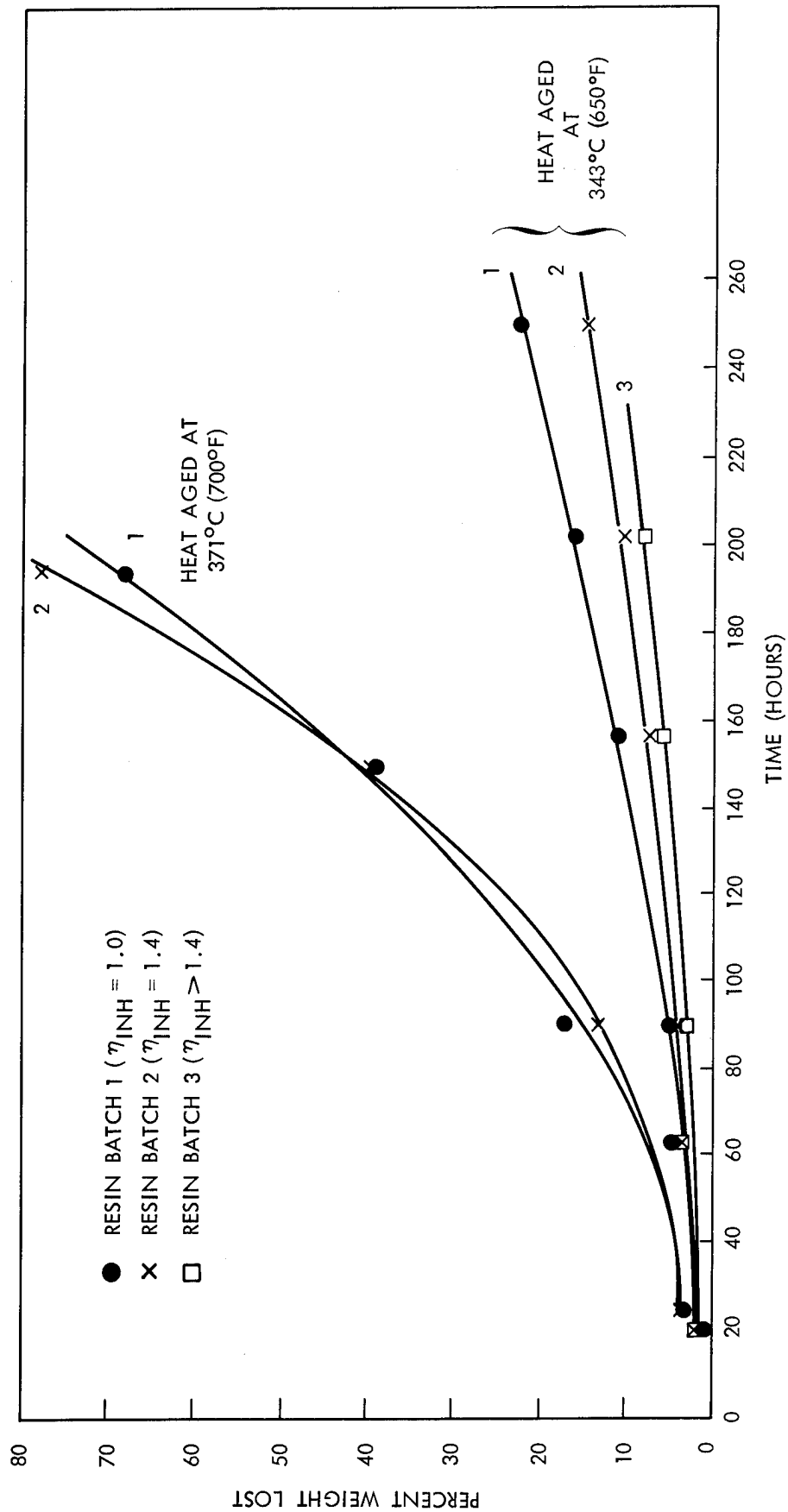

FIG. 10 LOSS OF WEIGHT ON HEAT AGING OF PPQ-1 SOLID RESIN POWDERS

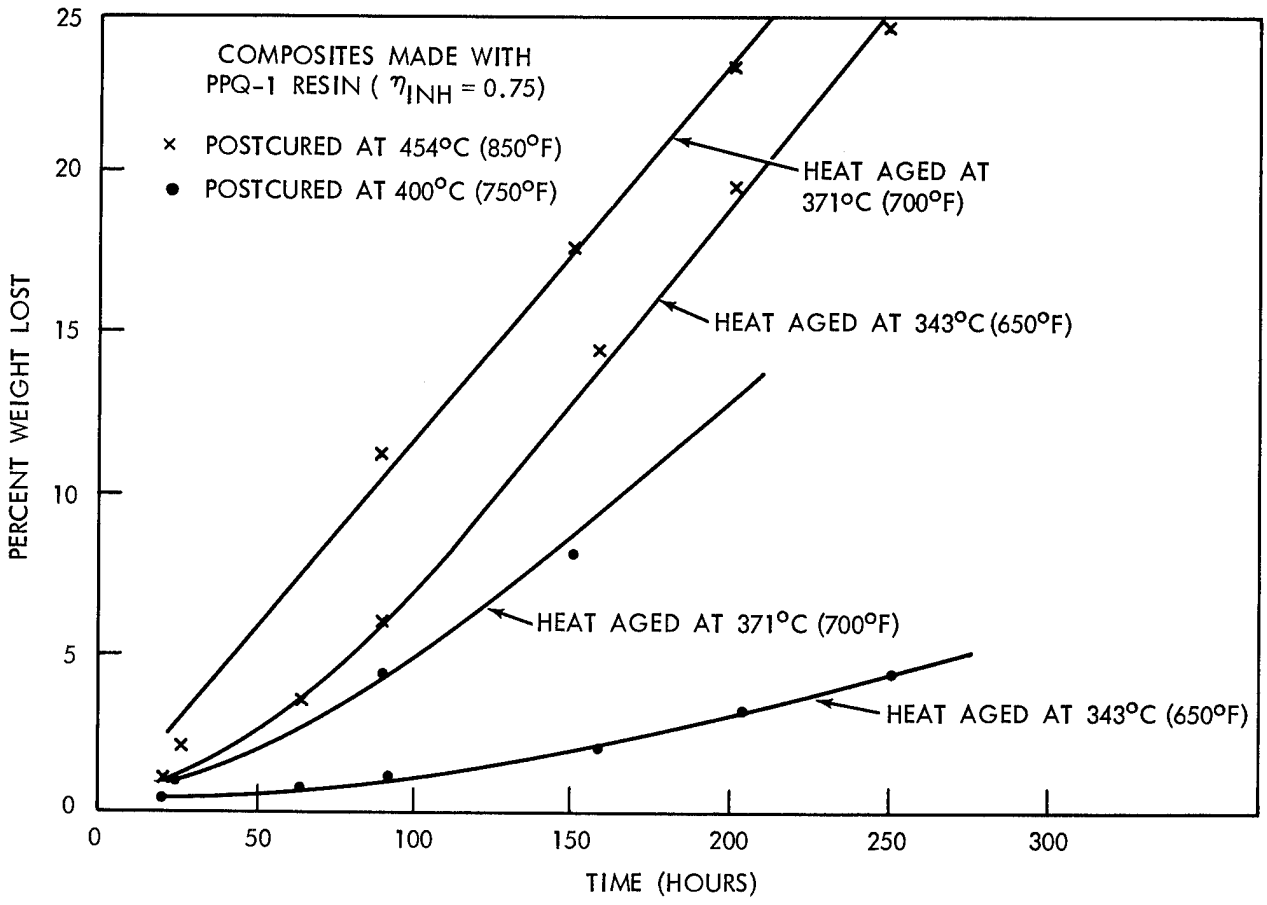

FIG. 11 LOSS OF WEIGHT ON HEAT AGING OF COMPOSITE PIECES FROM PANEL SET #1

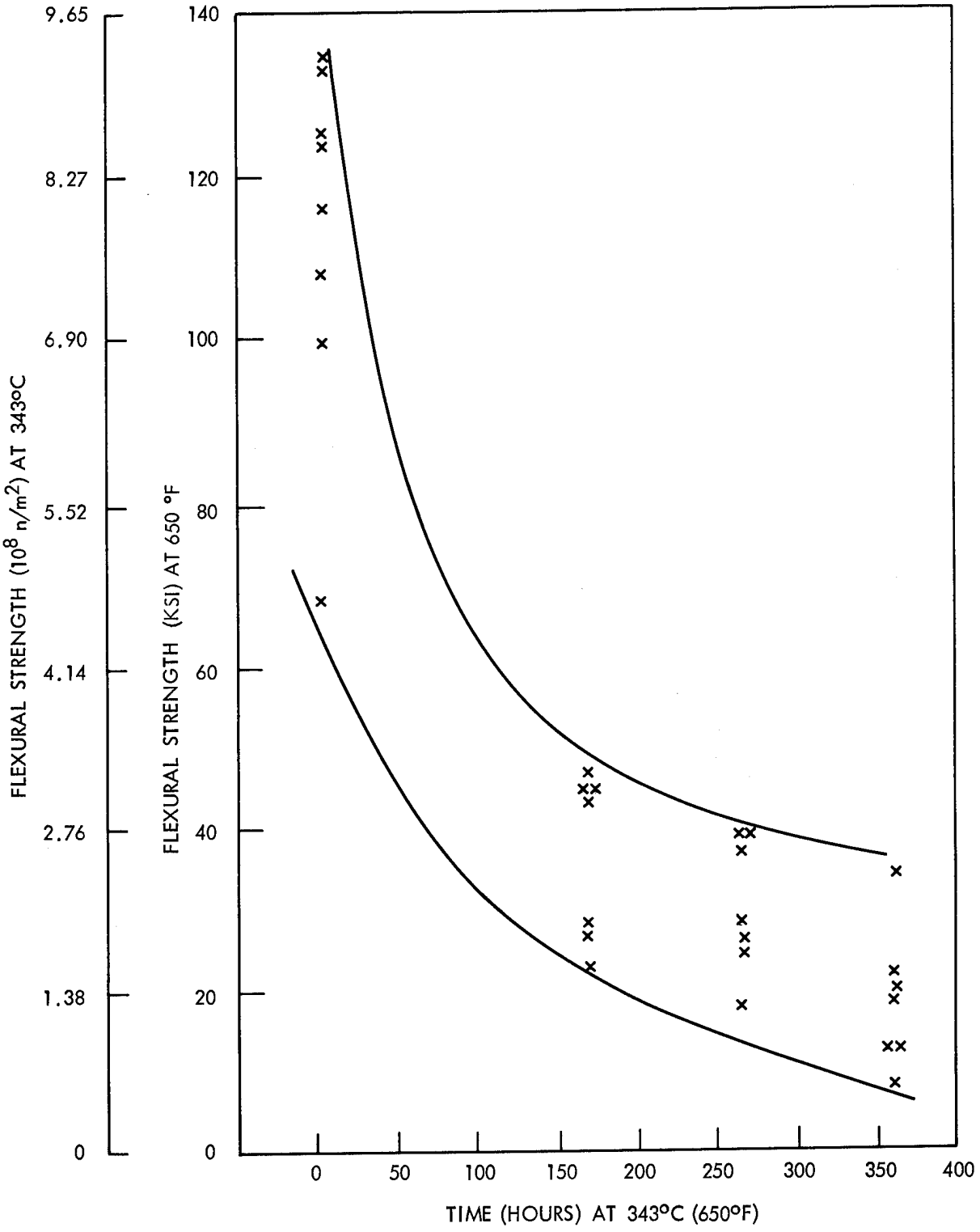

FIG. 12 FLEXURAL STRENGTH RETENTION AT 343°C VS. HEAT AGING TIME AT 343°C

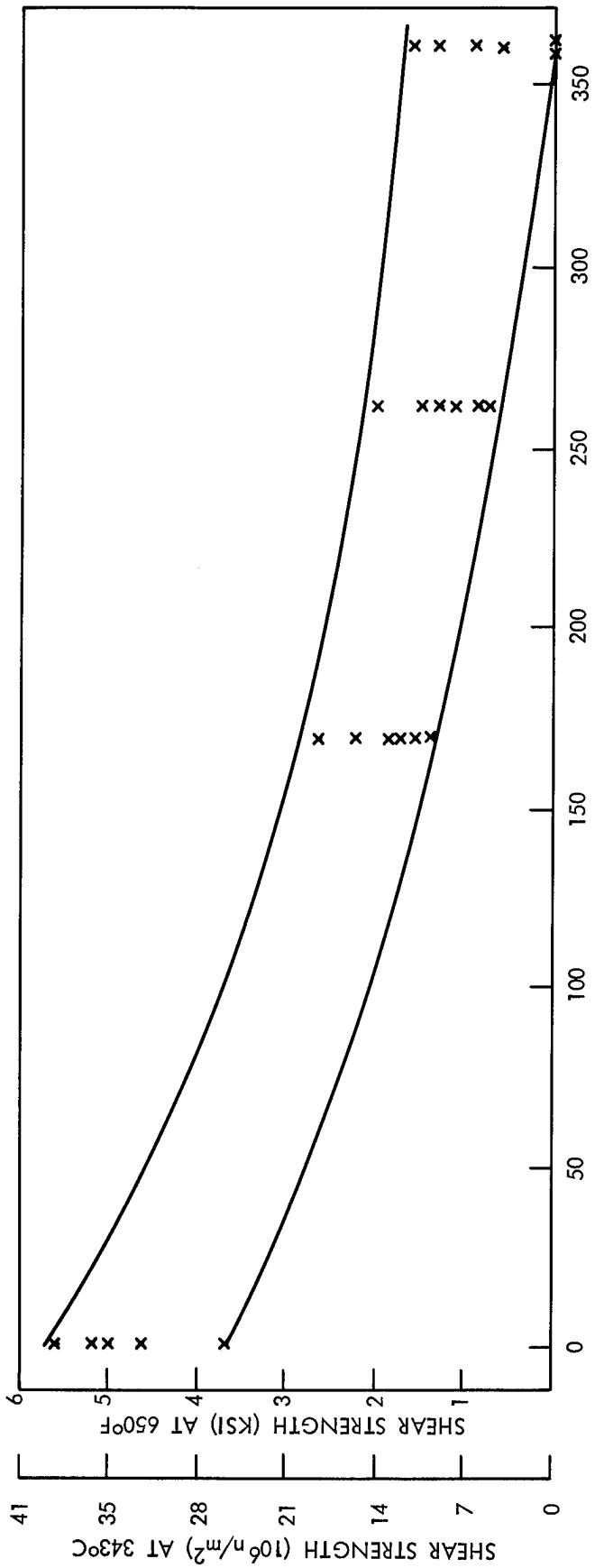

FIG. 13 INTERLAMINAR SHEAR STRENGTH RETENTION AT 343°C VS. HEAT AGING TIME AT 343°C.

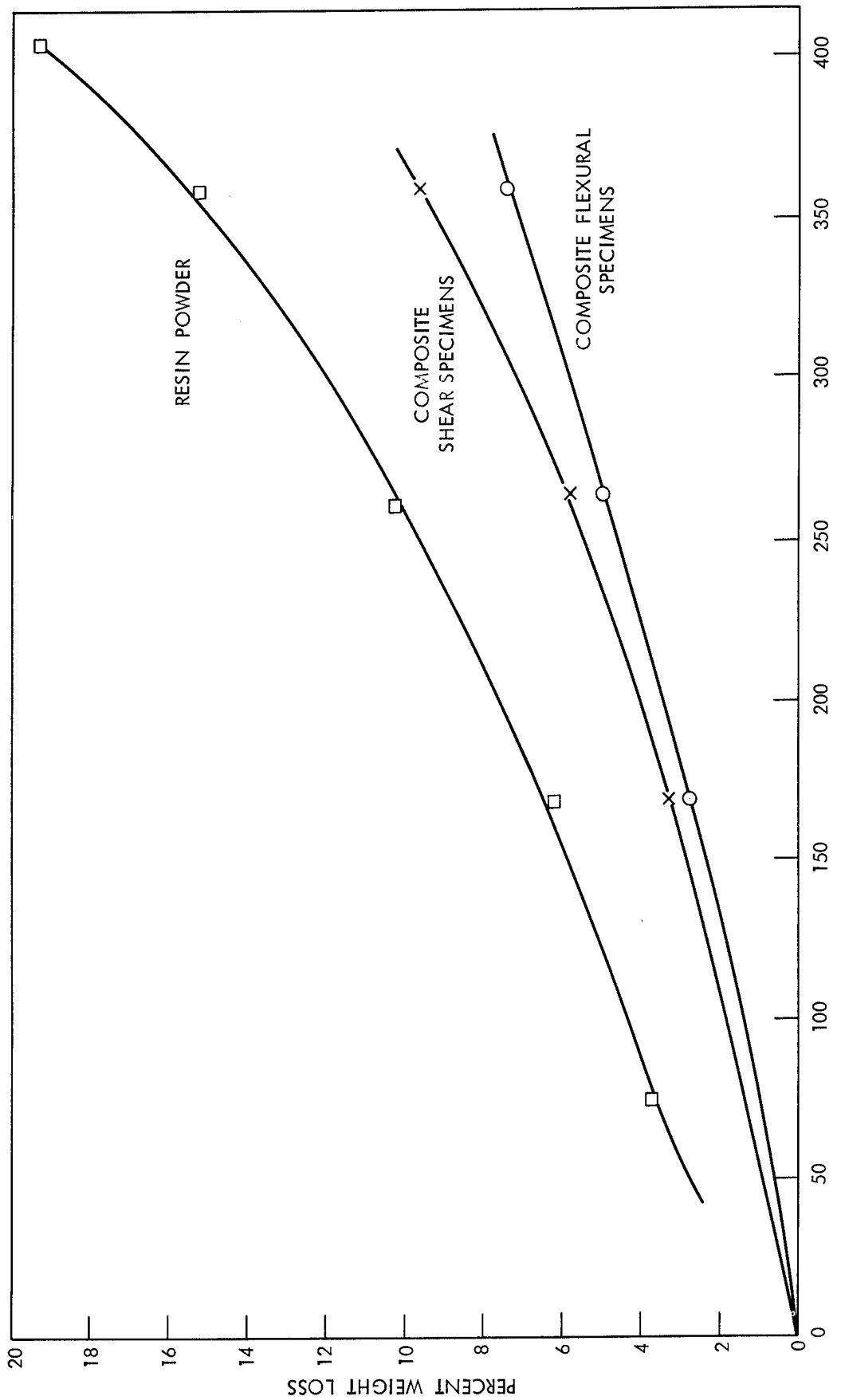

FIG. 14 HEAT AGING WEIGHT LOSS OF PPQ CARBON COMPOSITE SPECIMENS AND RESIN POWDER, PANEL SET #2

DISTRIBUTION

	Copies
Commander, Naval Air Systems Command U. S. Navy Department Washington, D. C. 20360 AIR-5203 (P. Goodwin) AIR-52032 (P. Stone) AIR-52032A (C. Bersch) AIR-52032C (J. Gurtowski) AIR-52032D (M. Stander) AIR-320A (T. Kearns) AIR-604 (Library)	2
Commander, Naval Ordnance Systems Command U. S. Navy Department Washington, D. C. 20360 ORD-033 (B. Drimmer) ORD-0333 (S. Matesky) ORD-0333A (M. A. Kinna) ORD-9132 (Library) ORD-047 (H. B. Parker, B. S. Thomas)	2
Director, Deep Submergence Systems Project Office Naval Material Command Washington, D. C. 20360 Attn: PM-11221 (H. Bernstein)	
Commander, Naval Ship Engineering Center Prince Georges Center Hyattsville, Maryland 20782 Code 6101E (J. Alfers) Code 6101E03 (W. Graner)	
Chief of Naval Research 800 Quincy Street Arlington, Virginia 22217 Code 470 (Dr. John H. Shenk) Code 472 (H. W. Fox) Code 425 (Dr. S. Berman)	

Copies

Director, Naval Research Laboratory
Washington, D. C. 20390
Code 6050 (Dr. W. Zisman)
Code 6100 (Dr. R. Kagarise)
Code 8433 (Dr. I. Wolock)

Commander, Naval Weapons Center
China Lake, California 93555

Commander, Naval Undersea Warfare Center
3203 E. Foothill Boulevard
Pasadena, California 91107

Commander, Naval Underwater Systems Center
Newport, Rhode Island 02844

Commanding Officer, U. S. Naval Weapons
Evaluation Facility
Kirtland Air Force Base
Albuquerque, New Mexico 87117
Attn: William Gordon

Director, Naval Avionics Facility
Indianapolis, Indiana 46202
Attn: Carl Ferguson
Superintendent of Materials Division

Commander, Naval Weapons Laboratory
Dahlgren, Virginia 22448
Attn: W. A. Mannschreck, Code EA

Commander, Naval Air Development Center
Warminster, Pa. 18974
Attn: H. Bowen (Aero Materials Dept.)
L. Ritter
C. A. Cassola

Director, Air Force Office of Scientific
Research
1400 Wilson Boulevard
Arlington, Va. 22209
Attn: SIGL

Director, Strategic Systems Project Office
Department of the Navy
Washington, D. C. 20360

	Copies
Director, National Aeronautics and Space Administration 600 Independence Avenue, S. W. Washington, D. C. Attn: B. Achhammer, Code RPM	
Commander, Harry Diamond Laboratories Washington, D. C. 20438	
Federal Aviation Administration Office of Super Sonic Development 800 Independence Avenue, S. W. Washington, D. C. 20590 Attn: E. W. Bartholomew (SS-110)	
Director of Development Army Material Command Gravley Point Washington, D. C. 20315 Attn: AMCRD	
Commanding Officer Picatinny Arsenal Dover, New Jersey 07801 Attn: Plastics Laboratory	
Plastic Technical Evaluation Center Picatinny Arsenal Dover, New Jersey 07801	
Commanding Officer, U. S. Army Mobility Equipment R&D Laboratory Fort Belvoir, Virginia 22060 Attn: Technical Document Center	2
Director, Air Force Materials Laboratory Wright-Patterson AFB, Ohio 45433 Attn: MAG MAN (R. T. Schwartz) MANC (Plastics and Composites Branch) MANE (R. L. Stout) MANF (J. E. Ross and H. Ezekiel) MATC (S. Litvak, Bldg. 16) MAAM (R. O. Hugher) MACM (George Peterson, H. Schwartz)	

Copies

Director, Forest Products Laboratory
Madison, Wisconsin 53705

University of Dayton
Research Institute
Dayton, Ohio 45409
Attn: Research Librarian

Commanding Officer, Army Materials and
Mechanics Research Center
Watertown, Mass. 02172
Attn: Library
Dr. Janet S. Perkins

Director, NASA-Langley Research Center
Spacecraft Materials Section
Langley Station
Hampton, Virginia 23365
Attn: Dr. Norman Johnston

Advanced Composites Information Center
Lockheed-Georgia Company
Department 72-14, Zone 402
Marietta, Georgia 30060

Whittaker Corporation
Research and Development Division
3540 Aero Court
San Diego, Calif. 92123
Attn: Dr. W. Fitzgerald

UNCLASSIFIED

Security Classification

DOCUMENT CONTROL DATA - R & D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY *(Corporate author)*
Naval Ordnance Laboratory
Silver Spring, Md. 20910

2a. REPORT SECURITY CLASSIFICATION
Unclassified

2b. GROUP

3. REPORT TITLE
FABRICATION AND TESTING OF CARBON FIBER/PHENYLATED POLYQUINOXALINE RESIN COMPOSITES

4. DESCRIPTIVE NOTES *(Type of report and inclusive dates)*
Techniques developed for fabricating PPQ composites and resulting composite strength. July 1969 - June 1971

5. AUTHOR(S) *(First name, middle initial, last name)*
Mary Lynda Santelli

6. REPORT DATE	7a. TOTAL NO. OF PAGES	7b. NO. OF REFS
18 November 1971	41	18

8a. CONTRACT OR GRANT NO.	9a. ORIGINATOR'S REPORT NUMBER(S)
b. PROJECT NO.	NOL TR 71-187
c.	9b. OTHER REPORT NO(S) *(Any other numbers that may be assigned this report)*
d.	None

10. DISTRIBUTION STATEMENT
Approved for public release; distribution unlimited

11. SUPPLEMENTARY NOTES	12. SPONSORING MILITARY ACTIVITY
None	Naval Ordnance Laboratory Silver Spring, Md. 20910

13. ABSTRACT

Techniques were developed for fabricating carbon fiber composites using a Naval Ordnance Laboratory developed high temperature resin, phenylated polyquinoxaline (PPQ). Composites with interlaminar shear and flexural strengths about 80% of those for similar epoxy composites were obtained. Heat aging at 343°C (650°F), in air, revealed excellent initial strength retention at temperature but significant loss of strength after 168 hours.

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Carbon Fiber Composites Phenylated Polyquinoxaline Resin Composite High Temperature Resins Composite Fabrication Techniques						